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A COMPARISON OF THE BROMINATION DYNAMICS OF PITCH-BASED
AND VAPOR-GROWN GRAPHITE FIBERS

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SUMMARY

The electrical resistance of pitch-based P-100 fibers (Union Carbide) and experimental organic vapor-grown fibers (General Motors, GA Technologies, and University of Nebraska) was recorded in-situ during bromination and subsequent exposure to ambient laboratory air. The results of this study indicate that the bromination and debromination reactions proceed much slower for vapor-grown fibers than for pitch-based. While this may be due in part to the larger diameter of the vapor-grown fibers, the majority of the effect can probably be attributed to the differences in graphene plane orientation between the fiber types. Although the reactions are slower in the vapor-grown than in the pitch-based fibers, the extent of reaction as measured by the change in electrical resistance is essentially the same, with comparable (or larger) decreases in resistivity. The bromination reaction proceeds with one or more plateaus in the resistance versus time curves, which suggests staging and strengthens the argument that these fibers produce true intercalation compounds.

INTRODUCTION

The aerospace industry has an evergrowing need for high-strength, low mass density, electrically conducting materials. While aluminum has been the material of choice in the past, the trend is to consider the lower density, higher strength carbon or graphite fiber/epoxy matrix composites. Carbon fibers and, to a lesser extent, graphite fibers, have the distinct disadvantage of having only moderate electrical conductivity, a problem that is further exacerbated by the nonconducting matrices in which they are usually embedded. One approach to improving the conductivity of graphite fibers is via intercalation; that is, the insertion of guest molecules between the graphene planes. Conductivities comparable to copper have been obtained in graphite crystals using this technique (ref. 1).

Unfortunately, graphite intercalation compounds exhibiting the highest conductivities react with air and/or moisture, which severely limits their applicability. Recently it has been shown that when bromine is used as the intercalate and pitch-based P-100 fibers (Union Carbide) are used as the host, the resulting compound has a resistivity comparable to that of stainless steel ($50 \mu\Omega\text{-cm}$). Furthermore, the compound is stable in air, vacuum, high humidity, and temperatures up to 200°C (ref. 2).

An attractive feature of bromine as an intercalate is that the reaction occurs at room temperature without catalyst. Jaworske (ref. 3) has analyzed the dynamics of this reaction as a function of bromine vapor pressure and found that below a bromine pressure of 100 torr the reaction will not occur, but above that pressure the extent of reaction is not a function of bromine

pressure (although the rate, of course, was). In the study presented here, both the rate and the extent of reaction were examined at room temperature as a function of fiber host.

METHODS AND MATERIALS

Fibers used in this study were pitch-based P-100 fibers manufactured by Union Carbide. They were heated at 350 °C for 24 hr in air to remove a polyvinyl alcohol sizing which is added to improve their bulk handling characteristics. Also used were benzene-grown fibers grown by GA Technologies (GA) (ref. 4), and natural-gas-grown fibers developed and provided by G.G. Tibbetts at General Motors Research Laboratories (GM) (ref. 5), and by J.A. Woollam at the University of Nebraska, Lincoln (UNL) (ref. 6). Both the benzene-grown and natural-gas grown fibers are grown by a process of chemical vapor deposition and will be referred to as vapor-grown fibers. They were used as received.

There are many differences between the pitch-based and vapor-grown fibers. One of the most important differences to potential users is that P-100 fibers are extruded and as such are available in spools of continuous length. This makes them suitable for high-strength applications and allows the use of rapidly developing filament winding techniques. They can also be woven into fabrics and resin preimpregnated mats, or can be chopped to any convenient length. In contrast, the vapor-grown fibers are grown by a chemical vapor deposition process and as such are limited in their length by the size of the reaction furnace (a few centimeters at present). Thus, the vapor-grown fibers are suitable only for chopped fibers applications. A second difference is that while P-100 fibers excel in high tensile modulus and strength (ref. 7), the vapor-grown fibers excel in electrical and thermal conductivity (ref. 8).

There are also differences among the three kinds of vapor-grown fibers. The GA fibers were grown by the method of Endo (ref. 9) and benzene was used as their precursor. Both the GM and the UNL fibers were grown from methane by the method of Tibbetts (ref. 5). The heat-treatment temperatures also differ somewhat. Some of the properties of the fibers used in this study are shown in table I.

It should be noted that the vapor-grown fibers are still experimental and are grown in small batches. There may be considerable variation from batch to batch both in growth conditions and postproduction heat-treatment temperatures. Additionally, many of the vapor-grown fibers were strongly tapered. It was not unusual to have fibers which narrowed from 50 μm down to as little as 15 μm over a 1 cm length.

To monitor its resistance change during bromination a fiber was attached with carbon paint (Structure Probe Inc.) to either four platinum wires or to a four-point-probe chip made up of platinum sputtered on aluminum oxide (ref. 10). If a chip was used it was then attached to four platinum leads which fed through a glass reaction vessel (ref. 11). Fibers which were mounted onto the chips could be removed from the reaction vessel without disturbing the fibers. Fibers mounted directly onto the platinum wires were removed and transferred

to copper on IC board four-point-probe chips. In this way the long-term debromination behavior of the fibers could be monitored conveniently.

The resistance was measured by the four-point technique with the current supplied to the outer leads by a Keithley Model 220 constant current source, while the voltage across the inner leads was monitored by a Keithley Model 181 nanovoltmeter. Both instruments were under the control of an IBM PC/XI computer. Before each data point was taken, the current was turned off, and the voltmeter was zeroed to eliminate spurious voltages. Data were recorded digitally at 15 sec intervals during the course of the reactions.

After a pristine fiber baseline resistance was established, the mounted fiber was lowered into a large test tube filled with bromine vapor. The vapor was in equilibrium with several milliliters of bromine liquid at room temperature, so its pressure was approximately 165 torr. The fibers become more conductive as they react with the bromine. The change in the resistance of the fibers was used to characterize the extent of reaction. The reaction was assumed to be complete when the resistance stopped its downward trend and leveled out. After that point the probe containing the fiber was removed from the bromine vapors and placed in a stream of ambient laboratory air. When the resistance reached a new equilibrium value, the fiber was removed from the probe and stored at ambient laboratory conditions. The fibers were checked periodically for further changes in their resistance.

RESULTS AND DISCUSSION

Bromination

A plot of the resistance ratio (resistance, R /initial resistance, R_0) of six P-100 fibers immersed in the bromine vapor is shown in figure 1. There are three distinct regions of note in the curve, implying three distinct aspects of the reaction which dominate at different times. During the first 70 to 100 sec there is a very sharp drop in the resistance with time until the resistance ratio is about 0.7. This is followed by a period of slower reaction which lasts for about 40 min and which is very complex in functional form. This period ends abruptly when the resistance ratio reaches a value of about 0.06. Last comes a region of equilibrium with the bromine vapor which shows no substantial change even after several hours. The results presented in figure 1 are qualitatively similar to those of Jaworske and Zinolabedini (ref. 12).

Plots of resistance ratio versus time in the bromine for the GA and UNL fibers are shown in figures 2 and 3, respectively. The first characteristic to note is that the time scale for reaction is much longer for these fibers. The three regions observed in the pitch fibers are also seen in both the GA and the UNL fibers, though the functional form in the middle region varies from each other and from the P-100. Again there is a region lasting about 60 to 100 sec where the GA and UNL fiber resistance ratios drop to about 0.9. The second, more complex region is much longer for the vapor-grown fibers than for the pitch-grown fibers, stretching out from about 3 to 11 hr. There were several discontinuities in the slope of the resistance ratio versus time graph that are strongly suggestive of staging (see fig. 4). They are particularly apparent in the fibers with the longest reaction times. Once again these

resistance drops end abruptly at a nearly constant value of 0.35 for the GA fibers and 0.1 for the UNL fibers. No subsequent resistance change was observed even after several hours.

The bromination reaction of the GM fibers appears qualitatively different from the rest of the fibers sampled (see fig. 5). Once again there is a sharp drop in the resistance in the first minute of reaction to about 75 percent of the initial resistance. But this is followed by a smoothly declining rate of reaction which lasts about 1 hr. There are no features suggestive of staging in this reaction, and it does not end abruptly. Furthermore, the minimum resistance ratio is only about 0.6. Although the GM fibers used in this study had resistivity drops only in the order of 60 percent, GM fibers tested in the past have had resistivity drops to about 12 percent of their original value.

Rather than comparing the details of reaction rates of individual fibers (which showed considerable variation), the broad reaction trends will be examined. This will be done with an eye towards explaining the trends in terms of fiber structure.

Figure 6 shows the average resistance ratio as a function of time for each of the four fiber types tested. Note that the initial drop in the resistance ratio is much greater for the pitch fiber than it is for the vapor-grown fibers. This could be explained if the initial drop is dominated by the absorption of bromine onto the surface of the fiber. The surface of the pitch fiber contains the edges of the graphene planes and so is microscopically porous (fig. 7). The vapor-grown fibers, on the other hand, have the faces of the graphene planes parallel to the surface, showing a tighter surface density of carbon atoms (fig. 8). In addition, the vapor-grown used in this study had larger diameters than the pitch-grown fibers, so their surface to volume ratios were lower. This would imply a smaller percentage drop if the resistance change is dominated by a surface reaction.

Following the sharp initial resistance drop is a region of steadily declining resistance which varied with the fiber type and indeed with individual fiber. It is this region which determines the total bromination time. While there is an undoubtedly important component to the reaction that involves diffusion of the bromine through the fiber, attempts at curve fitting revealed that the functional form is far too complex to suggest that the process is diffusion controlled. Discontinuities appear in this region which suggest the formation of discrete stages. The GA and P-100 fibers have one large plateau, while some of the UNL fibers may have as many as four. Assuming the final intercalation compound is stage 2 (after the studies using single crystals), the large plateau in the bromination reactions might correspond to the stage 3 to stage 2 transition.

This complex portion of the reaction ends rather abruptly, probably signaling that the bromine has reached saturation to the core of the fiber. This is supported by observations which show that fibers which are incompletely brominated appear to have regions of bromination and regions of pristine fiber, rather than a generally more dilute bromine concentration (ref. 13). This third region is then a generally level portion of the reaction with only a slow decline in resistance as final equilibrium is reached.

The decrease in resistivity for fibers in equilibrium with bromine appears to be less for vapor-grown fibers than for the pitch-based. P-100 fibers in equilibrium with bromine have an average decrease in their resistance by a factor of 16, whereas UNL, GA, and GM fibers have resistance decreases of only 10, 5, and 1.6, respectively. There are experimental difficulties which may lead one to question these final values, as will be discussed below.

The microscopic geometry of the pitch-based P-100 fibers is radically different from that of the vapor-grown fibers. The P-100 fiber has its graphene planes oriented radially, like the spokes of a wheel (fig. 7). This facilitates the insertion of the bromine into the spaces between the planes and explains the fast bromination dynamics. In contrast, the vapor-grown fibers have their graphene planes oriented circularly, like the growth rings of a tree (fig. 8). In order to penetrate the fiber, bromine must enter through microcracks and defects along the surface of each plane. This results in a much slower reaction.

There exists some controversy as to whether the bromine is really intercalated into these graphite fibers or merely adsorbed onto the surface and into the grain boundaries. The controversy exists because no fiber x-ray diffraction pattern has been observed which unambiguously shows an increase in the crystallographic "c" axis. There are reports in the literature of "c" axis expansions on the order of 0.02 Å, based on the 002 peak (ref. 14), but the peaks obtained from fibers are so broad as to cast doubt on increases of this magnitude.

The stepwise decline in the resistance of the fibers upon bromination, while not conclusive, is strongly suggestive of a true intercalation reaction. There are two additional pieces of evidence that lead one to suspect that the fibers form true intercalation compounds. The first is the existence of a threshold pressure. The resistivity of P-100 fibers is not effected by bromine below a pressure of about 100 torr (ref. 3). While threshold pressures have been observed in many different intercalation compounds (see, for example ref. 15), adsorption occurs at even the lowest pressure (ref. 16). The second is that intercalation has been reported for dozens of graphite forms, many of which are less crystalline than these fibers (ref. 14). By analogy one would expect the reaction with bromine to be intercalation.

Debromination

When graphite fibers are removed from the bromine atmosphere and allowed to equilibrate with ambient laboratory air, their resistance increases until it reaches an equilibrium value which is above that of the fiber in bromine vapor, but below the pristine resistance. Figure 9 shows the average resistance ratio (resistance, R /resistance of fiber in bromine vapor, R_{\min}) for the P-100 fibers. The figure shows that the ratio jumps to approximately 2.7 within the first two minutes. It then drifts slowly upwards for about 2 hr until it levels off at a value near 3.1. This final value is about a factor of 5.0 less than the pristine resistance. There are no discontinuities observed in the debromination reaction for each fiber. The apparent discontinuity observed in figure 9 probably occurs as a result of averaging

resistance anomalies associated with contact problems encountered while removing the fibers from the bromine.

The corresponding study on the vapor-grown fibers (UNL) is also shown in figure 9. Note that behavior similar to that of the P-100 fibers is observed, but on a much longer time scale. The time it takes these fibers to come to equilibrium is on the order of eight days. Similar behavior is observed in the debromination reaction of the vapor-grown fibers from GM.

When the GA fibers were exposed to air after bromination, quite different behavior was observed. The resistance of the fibers dropped abruptly to a value of about 0.9 that of the fibers in the bromine vapor and then remained constant. This is believed to be an artifact, but warrants further investigation.

The micro-structure of these fibers suggests why the debromination dynamics should be slower for the vapor-grown fibers than the pitch-grown. The radial geometry of the pitch fiber implies that the bromine can travel between the graphene planes in a relatively unobstructed path from the interior of the fiber to the surface. Bromine trapped in the circular geometry of the graphene planes in the vapor-grown fibers must diffuse through the planes via defects, or wind its way along grain boundaries.

This simple picture, however, does not explain why a substantial amount of the bromine does not diffuse out of the fibers. Brominated then debrominated P-100 fibers are fully 18 percent by weight bromine (ref. 17), and study shows that the bromine is not randomly distributed. In fact, there is no bromine on the surface in concentrations detectable by Auger analysis or by EDS (ref. 12). The bromine is instead distributed through the cross section of the fiber in a nearly Gaussian manner (ref. 18).

Do the debromination dynamics shed light on whether the bromine is intercalated into the fiber or adsorbed onto grain boundaries? The rapidity with which the pitch fiber resistance increases favors an adsorption mechanism but does not rule out intercalation. HOPG is known to intercalate bromine and yet rapidly loses most of its bromine when removed from the vapor. One would expect to see discontinuities arising from the destaging as the sample degases in an intercalation compound. This is not observed. However, the peculiar manner in which the bromine is distributed in the degassed fiber; that is, only in the center, implies that this is not a reversible reaction. Perhaps then, one would not expect to see reversible dynamics.

The final values for the improvement in the resistance of these fibers are very similar. P-100 fibers which have been brominated and then allowed to equilibrate with the air have their resistance lowered by a factor of 5.0. Corresponding values for the GM, GA, and UNL fibers are 1.3, 5.5, and 8.0, respectively. This seems to contradict the assertion made above that the extent of reaction is less for the vapor-grown fibers.

Which, then, is the more accurate indicator of the extent of reaction, R_0/R_{min} or R_0/R_{final} ? When the fiber is submerged under bromine gas, the possibility exists for many complicating chemical reactions. There are significant resistance changes in a platinum wire measured under identical conditions. While the four-point method of resistance measurement should negate

these differences, erratic resistance measurements were not uncommon. It was noted that the vapor grown fibers seemed especially susceptible to this erratic behavior. For these reasons the debrominated fiber resistance data are considered more reliable. Thus, the extent of reaction was probably similar among the P-100, GA, and UNL fibers. Further study is under way to clarify this point. It should be noted that while the GM fibers apparently did not react to the extent of the other fibers, previous batches of GM fibers have reacted to a similar extent (as judged by their initial and final resistivities).

CONCLUSIONS

A summary of the findings of this study is contained in table 2. Because of the variations observed from fiber to fiber within a batch, and from batch to batch within a fiber type for the vapor-grown fibers, one must use caution when drawing conclusions. The following conclusions concerning the bromination reaction, however, seem unmistakable. First, the rates of the bromination reaction are slower for vapor-grown fibers than for pitch-based fibers. This may be due in part to the larger size of the vapor-grown fibers, but the majority of the effect can probably be attributed to the differences in graphene plane orientation. Second, the extent of reaction, as measured by the resistance ratio R_0/R_{min} , appears to be less for the vapor-grown fibers than for the pitch-based. If, however, the final resistance ratio R_0/R_{final} is used as the indicator, comparable (or larger) decreases in resistivity than pitch-based fibers are observed. Third, the intercalation process occurs with the presence of one or more discontinuities in the resistance curves, which suggests staging and strengthens the argument that these fibers produce true intercalation compounds.

There are also three related conclusions concerning the debromination dynamics. First, the deintercalation rates are slower in the vapor-grown fibers than in the pitch-based. Second, deintercalation occurs without discontinuities in the resistance values. And last, although the deintercalation rates are also slower for the vapor-grown fibers, a stable residue compound with a significant decrease in the fiber resistivity is the final product.

Thus, the bromination and debromination processes for the vapor-grown fibers are factors slower for the same reaction in pitch-based fibers. Despite the differences in the bromination and debromination dynamics, the characteristics of the final products, both in and out of the bromine environment, are very similar.

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TABLE I. - PROPERTIES OF GRAPHITE FIBERS USED
IN THIS STUDY (REFS. 4 TO 8)

Type	Diameter, μm	Resistiv., $\mu\Omega\text{-cm}$	Thrm. Cnd., W/m-K	Modulus, GPa	Strength, GPa	Heat Tre, $^{\circ}\text{C}$
P-100	10	250	520	2.2	690	3000
GM	15-50	100	1200	1.0	200	2800
GA	20-40	90	1300	---	---	3000
UNL	15-50	90	----	---	---	2950

TABLE II. - SUMMARY OF BROMINATION OF GRAPHITE FIBERS

Type	R_o/R_{min}	$R_{\text{final}}/R_{\text{min}}$	R_o/R_{final}	$\rho_{\text{final}},$ $\mu\Omega\text{-cm}$	Br time, ^a sec
P-100	16	3	5	50	2400
GM	1.6	1.2	1.3	77	3000
GA	5	0.9	5.5	16	6000
UNL	10	1.2	8	11	28000

^aTime for bromination reaction to come to equilibrium at 165 torr of Br_2 .

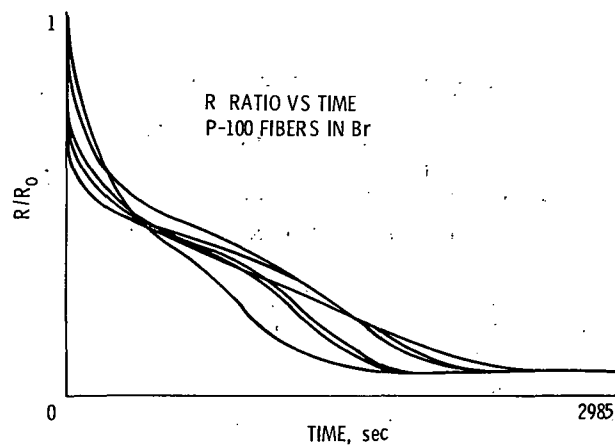


Figure 1. - The resistance ratio (R/R_0) of 6 P-100 graphite fibers plotted as a function of time exposed to 165 torr of bromine.

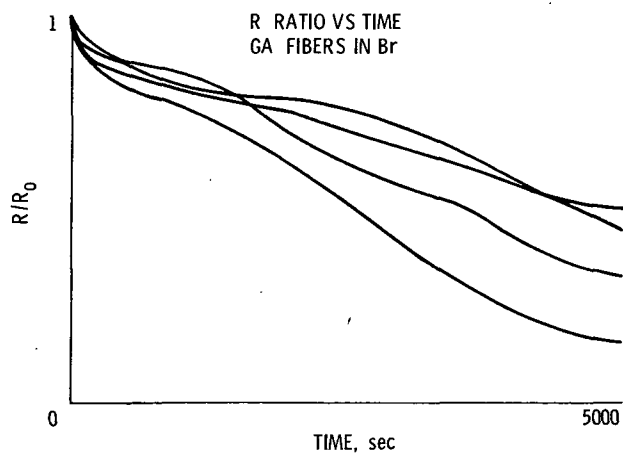


Figure 2. - The resistance ratio (R/R_0) of 4 GA graphite fibers plotted as a function of time exposed to 165 torr of bromine.

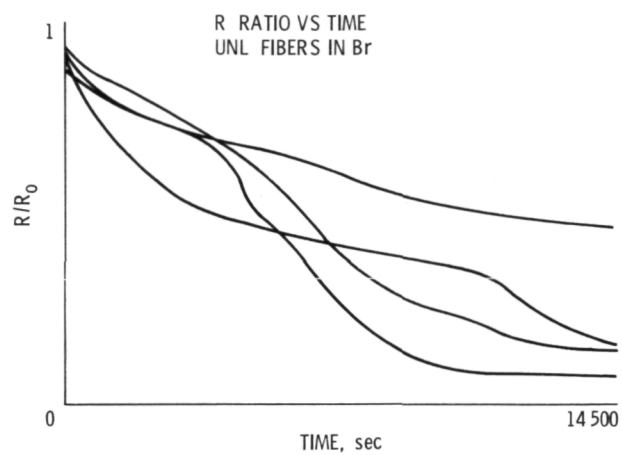


Figure 3. - The resistance ratio (R/R_0) of 4 UNL graphite fibers plotted as a function of time exposed to 165 torr of bromine.

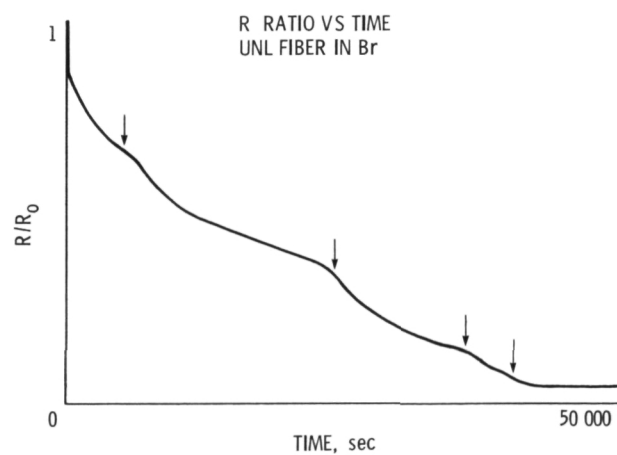


Figure 4. - The resistance ratio (R/R_0) of a UNL graphite fiber plotted as a function of time exposed to 165 torr of bromine. Note the discontinuities (indicated by arrows) which suggest stage transitions.

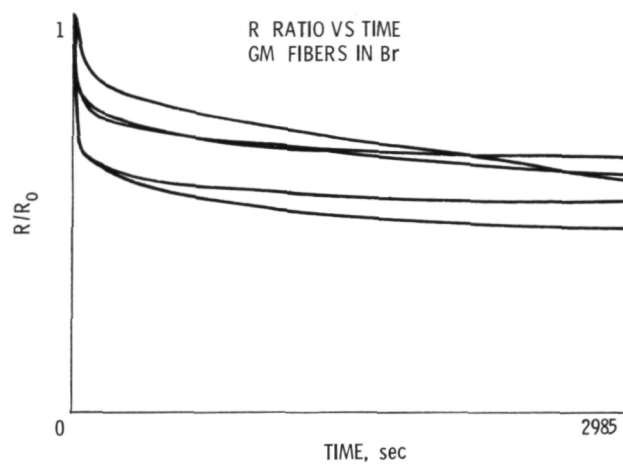


Figure 5. - The resistance ratio (R/R_0) of 5 GM graphite fibers plotted as a function of time exposed to 165 torr of bromine.

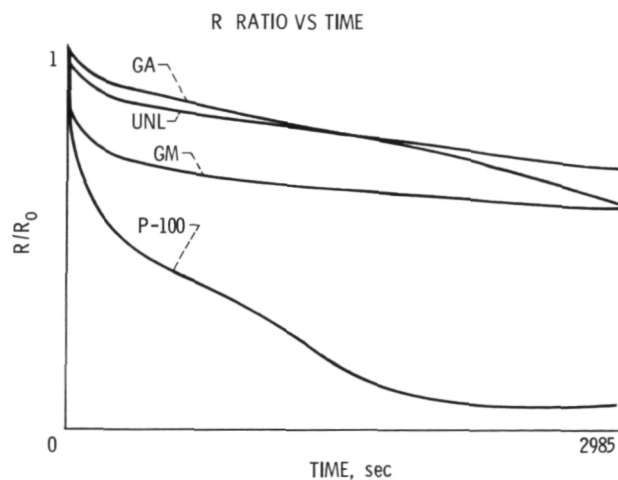


Figure 6. - The average resistance ratio (R/R_0) as a function of time exposed to 165 torr of bromine for P-100, GA, UNL, and GM graphite fibers.

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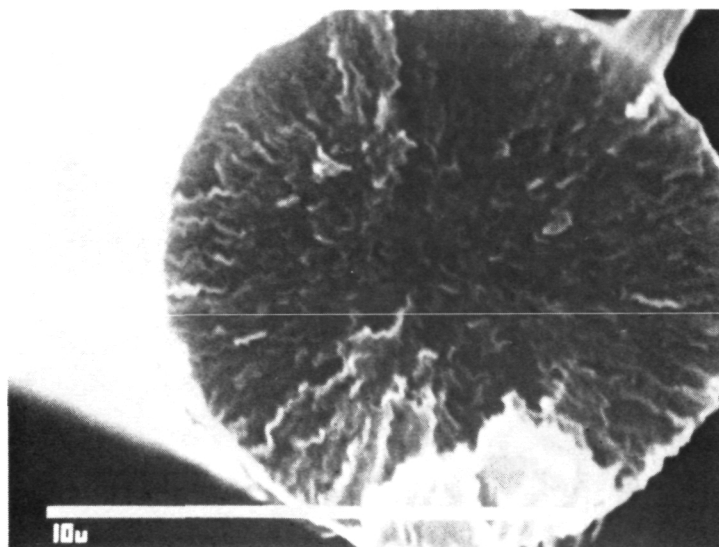


Figure 7. - Scanning electron micrograph of a P-100 graphite fiber showing the radial orientation of the graphene planes.

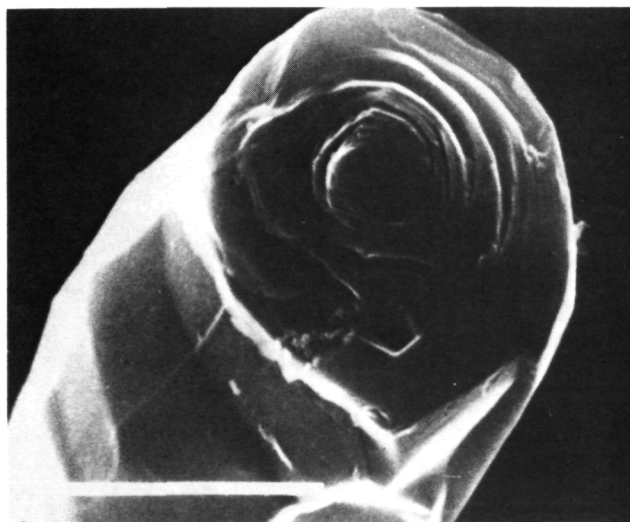


Figure 8. - Scanning electron micrograph of an organic gas derived graphite fiber showing the circular orientation of the graphene planes.

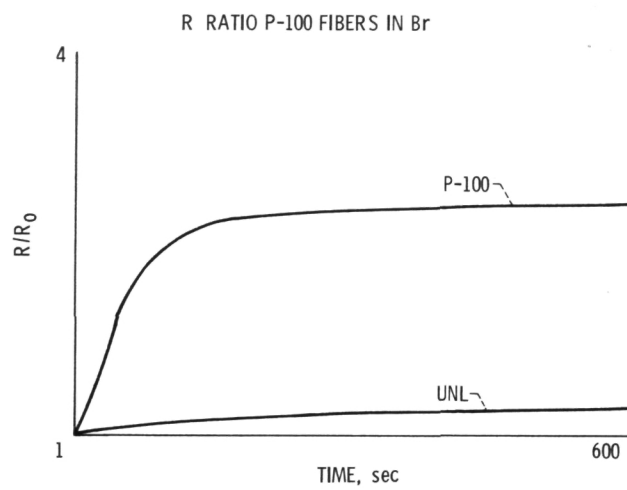


Figure 9. - The average resistance ratio (R/R_0) of 4 P-100 and 3 UNL graphite fibers as a function of time since removed from bromine into air.

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